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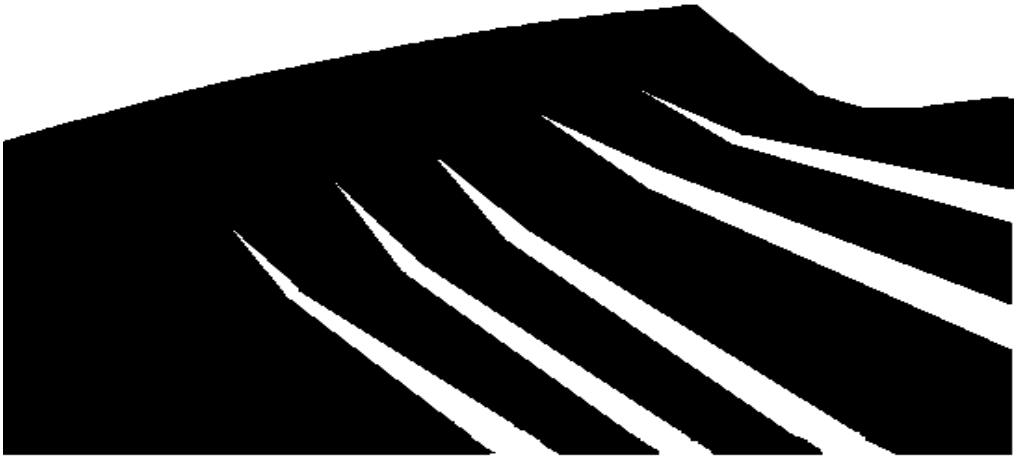
ZEOLITE PURIFICATION/SEPARATION PROCEDURE

LOS ALAMOS

QUALITY PROGRAM



APPROVAL FOR RELEASE	
S. J. CHIPERA - PREPARER Signature on file	DATE Date on file
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Los Alamos
Yucca Mountain Site Characterization Project

HISTORY OF REVISION

REVISION NO.	EFFECTIVE DATE	PAGES REVISED	REASON FOR CHANGE
R0 - R1	N/A	N/A	History of revisions not in place.
R2	07/11/91	3, 4, & 6	Change of responsibilities. Sections added on the detection of malfunctions, safety considerations, and preparatory verification – hold points. Added steps to reflect that small amount of sample may also be processed using this procedure. Better defined the accept/reject criteria. Revision 1 of this procedure was previously identified as TWS-ESS-DP-110.
R3	02/14/97	All	Revised to comply with LANL-YMP-QP-06.3, requirements.

ZEOLITE PURIFICATION/SEPARATION PROCEDURE

1.0 PURPOSE

The purpose of this procedure is to describe the methods, procedures, and documentation used to purify / separate zeolites from zeolite-rich samples by sedimentation.

2.0 SCOPE

This procedure applies to zeolite samples purified / separated for the Yucca Mountain Project.

3.0 REFERENCES

LANL-YMP-QP-02.7, Personnel Training
LANL-YMP-QP-03.5, Documenting scientific Investigations
LANL-YMP-QP-17.6, Records Management
LANL-EES-DP-16, Siemens X-Ray Diffraction Procedure
LANL-EES-DP-25, Clay Mineral Separation and Preparation for X-Ray Diffraction Analysis.
LANL-EES-DP-101, Sample Speciman Collection, Identification and Control for Mineralogy-Petrology Studies

4.0 DEFINITIONS

None.

5.0 RESPONSIBILITIES

The following personnel are responsible for the activities identified in Section 6.0 of this procedure.

- The Principal Investigator (PI)
- Users of this Procedure

6.0 PROCEDURE

The use of this procedure must be controlled as follows:

- If this procedure cannot be implemented as written, YMP personnel should notify appropriate supervision. If it is determined that a portion of the work cannot be accomplished as described in this DP, or would result in an undesirable situation, that portion of the work will be stopped and not resumed until this procedure is modified, replaced by a new document, or the current work practice is documented in accordance with QP-03.5, Section 6.1.6.

- Employees may use copies of this procedure printed from the controlled document electronic file; however, employees are responsible for assuring that the correct revision of this procedure is used.

- When this procedure becomes obsolete or superseded, it must be destroyed or marked “superseded” to ensure that this document is not used to perform work.

6.1 Principle

Zeolite-rich samples can be purified or extracted from a sample by means of sedimentation in water. This method relies on the difference in particle sizes and densities of the mineral components in the sample. This method is preferred over other methods due to the low magnetic susceptibilities of the zeolite (magnetic separations) or where one is concerned with potentially altering the chemical composition of the zeolite (heavy-liquid density separations).

6.2 Equipment and Hardware / Software

- Shatterbox (or large ball mill) – most all acceptable

- Ultrasonic probe capable of ~200 W.

- No software is used.

6.2.1 Equipment Malfunction

N / A

6.2.2 Safety Considerations

The Ultrasonic probe is placed in a sound-dampening box to help protect the hearing of the operator.

6.2.3 Special Handling

None.

6.3 Preparatory Verification

6.3.1 Hold Points

None.

6.3.2 Calibration

Not applicable. Samples are examined for purity using X-ray powder diffraction in accordance with DP-16.

6.3.3 Environmental Conditions

The use of de-ionized or distilled water is necessary to ensure that no chemical alteration of the zeolites occurs due to cation exchange. Thoroughly clean all sample preparation equipment and work areas before use.

6.4 Control of Samples

6.4.1 Samples will be tracked, handled, shipped, and stored in accordance with DP-101.

6.4.2 Carefully label containers into which the sample separates will be placed. Label sides and top of each container so that tops cannot be switched.

6.5 Implementing Procedure

6.5.1 The zeolite-rich sample is first shatterboxed or ball-milled (1 to 2 minutes) to break the sample down to a particle size that is closer to the grain size of its constituent phases.

6.5.2 ~30 to 60 grams of the zeolite sample are placed in a 1000ml plastic beaker filled with ~700-800ml of deionized water. (Glassware, beakers, and settling times are to be scaled appropriately when working with amounts of sample smaller than 30-60 grams).

6.5.3 Disaggregate the sample for approximately 10 to 15 minutes at ~200 W using an ultrasonic probe. Longer times may be used if samples are difficult to disaggregate.

6.5.4 Place the sample on a vibrationally stable surface without cooling it (the ultrasonic probe generates heat in the suspension) and allow it to settle for 30 to 60 seconds. The sediment is composed of the coarse fraction (>~20um) which should include much of the quartz, feldspars, and other mineral impurities, and also any larger clumps of zeolites that were not disaggregated. This fraction may be discarded.

6.5.5 Decant or syphon the supernatant into a second beaker and allow it to rest on a vibrationally stable surface for ~1 hour to settle out the 20-30um size fraction. This is generally a fairly pure zeolite fraction.

6.5.6 Decant or syphon the supernatant into a third beaker and allow it to rest on a vibrationally stable surface overnight (15-20 hours) to settle out the ~3.0-1.0um size fraction. This should also be a rather pure zeolite fraction.

6.5.7 The remaining supernatant, although it may have a high concentration of zeolite remaining, generally has a high concentration of smectite. It can be discarded, or further processed to concentrate the smectite for clay mineral analysis using DP-25.

6.5.8 The sediments from 6.5.5 and 6.5.6 are dried by placing the beakers with the sediments on a warm surface such as a hotplate set at ~50°C or allowed to air-dry at room temperature.

6.5.9 The dried sediments may be X-rayed to determine their purity in accordance with DP-16. Steps 6.5.2 and 6.5.7 may be repeated if further purification / separation is desired.

6.6 Data Acquisition and Reduction

N / A

6.7 Potential Source of Errors and Uncertainty

- This technique requires that the initial sample be fairly rich in zeolite.
- Minerals intergrown with the zeolites (such as opal-CT in clinoptilolite) will not be separated.
- Two similar species of zeolites such as mordenite in clinoptilolite do not separate very well.

7.0 RECORDS

Records generated as a result of this DP are entries in laboratory notebooks or attachments to laboratory notebooks. The documentation should consist of any applicable items identified in Section 6.0 of this procedure. Laboratory notebooks should be kept in accordance with QP-03.5.

All records should be submitted to the Records Processing Center in accordance with QP-17.6.

8.0 ACCEPTANCE CRITERIA

8.1 Purity of the zeolite fraction may be confirmed by X-ray diffraction analyses. It is up to the certified personnel conducting the separation to decide whether the zeolite has been purified / separated sufficiently for the work to be conducted. This decision shall be documented in the YMP notebook of the certified personnel before conducting further research with the sample.

8.2 The notebook entry for a sample shall constitute evidence that the procedure has been implemented and satisfactorily accomplished for that sample.

9.0 TRAINING

9.1 Prior to conducting work described in Section 6.0, the user requires training to this procedure.

9.2 Training to this procedure is accomplished by “read only.” Training will be documented per QP-02.7.

10.0 ATTACHMENTS

None.